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(54) Title: METHOD FOR THE PREPARATION OF IMPLANTS MADE OF TITANIUM OR ALLOYS THEREOF

(57) Abstract

The invention relates to a method for treating the surface of surgical implants made of titanium or a titanium alloy, for instance dental implants, for implanting in bone tissue, in which method a rough surface is obtained. At the same time a cleaning action is obtained, should there be any contaminations. The method is characterized in that the surface is blasted with particles of an oxide of titanium, preferably titanium dioxide. In this way an implant having an improved retension in bone tissue is obtained.

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+ DESIGNATIONS OF "SU"

Any designation of "SU" has effect in the Russian Federation. It is not yet known whether any such designation has effect in other States of the former Soviet Union.

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METHOD FOR THE PREPARATION OF IMPLANTS MADE OF TITANIUM OR ALLOYS THEREOF.

Technical field of the invention

The present invention relates to a method for treating the surface of surgical implants, particularly dental implants, made of c.p. titanium or of a titanium alloy and intended to be implanted in bone tissue. One object of the invention is to achieve an implant having an improved retention in bone tissue by providing the implant with an improved surface structure. An additional object of the invention is to achieve an implant having a great degree of purity in regard of contaminating substances which may endanger the retention of the implant in the bone tissue. The invention is described below in connection with dental implants, but the invention can be adapted to the surface treatment of other surgical implants to be implanted in bone tissue.

Background to the invention

The most sensitive part in a dental implant system is the fixture, i. e. that part of the system which is screwed or inserted into a hole in the jaw bone and which thus comes into direct contact with the tissues in the jaw. A suitable material for these implants is titanium. The use thereof as a material in surgical implants was suggested early (for instance by Leventhal et al in the Journal of Bone and Joint Surgery vol 33A, No 2, April 1951).

It is known that the fixture has to be very clean if the ingrowth of the bone tissue onto the implant, the so called osseointegration process, shall function in an optimal way. It even has been maintained that a contamination corresponding to a monomolecular layer could endanger the continued existence of the implant in the tissue.

The structure of the surface of the implant also is important.

The Swedish patent SE-C-416175 (7902035-0) for instance states that a better result of an implant operation is achieved if an implant with a surface of titanium oxide is provided with pores having a size of 10 - 1000 nm defined as being "micropits" . This patent specification does however not clearly show how this surface differs from other known surfaces, for instance turned surfaces, or to what extent the retention of the implant is improved.

Several other publications have discussed the importance of the surface roughness for the retention of the implant, for instance the academic treatise by Jan Lundskog: "Heat and Bone Tissue" Laboratory of Experimental Biology, Department of Anatomy, University of Gothenburg, Sweden 1972, the article "Adhesion of bone to titanium", by S.G. Steinemann, J. Eulenberger, P.-A. Maeusli and A. Schroeder in Biological and Biomechanical Performance of Biomaterials, edited by P. Christel, A. Meunier and A.J.C. Lee, Elsevier Science Publishers B.V., Amsterdam 1986 and the article "Removal Torques for Polished and Rough Titanium Implants", Carlsson L., Röstlund T., Albrektsson B., Albrektsson T., Int J Oral Maxillofac Implants, 1988; 3:21-24. These publications indicate that it may be better with an implant surface having larger (macroscopic) irregularities or pores than those described in the above Swedish patent.

Implants have been made by cutting operations for a long time, primarily by turning. According to the above Swedish patent such a machining may result in a surface with microscopic irregularities. Several cleaning methods have been suggested and used, such as cleaning with organic solvents, electropolishing, sand blasting and treatment with alkalic and acid solutions.

One commonly used cleaning method thus is blasting with well known blasting agents, such as sand or aluminium oxide. Blasting with a blasting material having correctly sized particles results in the surface roughness which is desired in order to obtain a good mechanical retention in the bone. One problem with blasting the surface of the fixture is however that, although the existing contaminations may be removed, residues from the blasting agent may remain on the surface of the implant. The blasting agents commonly used, for instance aluminium oxide, thus may result in contaminations that are impossible or very difficult to remove in a subsequent cleaning process.

Description of the invention.

The object of the invention is to provide a treatment of implants made of titanium or titanium alloys which in one operation both ensures that the surface of the implant is clean and that the surface has the macroscopic structure which is necessary for a good retention of the implant in the jaw bone.

According to the present invention this is achieved in that implants of titanium or of an alloy of titanium are blasted with particles of an oxide of titanium, preferably titanium dioxide. Since titanium exposed to air has a chemically resistant layer of oxide, the most important one being titanium dioxide, the blasting operation will not introduce any foreign, contaminating material onto the implant surface, although effectively removing existing contaminations and providing the desired roughness.

In accordance with a preferred embodiment of the invention the oxide used for the the blasting comprises titanium dioxide.

In a further preferred embodiment of the invention, the oxide, which preferably comprises titanium dioxide, comprises particles having a grain size within the range of 1 - 2000 µm, more preferably particles having a grain size within the range of 1 - 300 µm. In another preferred embodiment the particles have a grain size within the range of 1 - 50 µm. Particles having a size being close to the upper limit preferably are used in the treatment of larger orthopaedic prostheses.

It has proved that the fixture obtains a surprisingly good force-transmitting capacity in implants whose surface have been treated in accordance with the invention. This is important since the fixture is subjected to considerable forces which are to be transmitted to the bone. The fixture therefore has to have a design which allows a safe transmission of these forces to the bone. As indicated above, the strength of the bond between the bone and the implant is improved if the surface of the fixture has a macroscopic roughness which is superimposed on the geometrical design of the fixture. The use of blasting particles having a size of 1 - 50 µm results for instance in a size of the irregularities on the surface of the implant in the range 1 - 25 µm (the size of the irregularities are normally smaller than the size of the particles). Smaller irregularities may of course also be superimposed on these irregularities. In this range of the size of the roughness, the strength of the bond implant - bone is considerably improved. It has been shown that this strength-improving effect is reduced if the size is reduced.

The strength-improving effect is negligible at sizes below 0.3 µm. Larger irregularities, 5 - 200 µm, result in an additional improvement of the strength of the bond implant - bone, but irregularities in the size range 100 - 200 µm may in some cases interfere negatively with the geometrical design of the implant. The size of the

irregularities here can be defined as an approximative diameter or width of a rounded respectively an oblong shape.

Examples

A. Fixtures made of titanium of the kind used by the applicant, AB Astra (Astra Meditec), were thoroughly cleaned and degreased in a conventional manner and were then blasted in a blasting equipment "Sandmaster, Ultrafine Sandblaster Type FG3-82, Wülsag, Zofingen, Switzerland". A titanium dioxide powder having a grain size of 10 - 53 µm was used ("Metco 102", Metco Scandinavia AB, Vårby, Sweden, normally used for plasma- or flame-spraying). Air was used as carrying medium in the blasting process. Each fixture was blasted about 20 seconds. The fixture was rotated and the blasting nozzle was moved up and down along the longitudinal axis of the fixture at an angle of about 90 degrees relative to the longitudinal axis of the fixture. The air pressure of the apparatus was set at 1 Bar (100kPa) above atmospheric pressure.

Some of the blasted fixtures were examined in an electron microscope. A rough, irregular surface was observed. The size of the irregularities were about 5 - 15 µm with superimposed irregularities having a smaller size.

The implants were then washed in organic solvents and a number of fixtures were examined by means of ESCA (Electron Spectroscopy for Chemical Analysis) in order to study the degree of cleanliness. The implants proved to be exceptionally clean.

B. The above experiment was repeated with another blasting equipment, "Abrasive Blaster, Mark 3", delivered by Belle de St Claire, 16147 Valerio St, Van Nuys, California 91406, USA. Blasting agent and carrying medium were the

same as above. The air pressure of the apparatus was set at 80 psi (5,5 bar, 550 kPa). Again a dental implant fixture from Astra Meditec was blasted. The blasting nozzle was moved axially up and down the surface of the implant about 5 - 10 mm away from the the surface. The fixture was simultaneously rotated. The nozzle then was moved over the outer lower end of the fixture. Duration about 20 seconds. Visually the fixture displayed a dull surface after the blasting. During an examination under a microscope it proved that the blasting had resulted in a rough surface.

Animal tests.

I. Histological analysis.

10 fixtures, which had been blasted in accordance with the above Example A, were operated into the upper jaw of Beagle dogs together with untreated control fixtures. A histological analysis after 2 respectively 4 months showed that the blasted implants had been osseo-integrated in the same way as the untreated control fixtures.

II. Summary of results obtained in animal studies relating to osseointegration and fixation strength of "smooth" respectively blasted surface of titanium implants.

The geometrical design and the surface (macro-, micro-structure) of an implant are two factors considered important for the osseointegration. In order to study these factors and the differences in the degree of the osseointegration and the strength of the fixation two studies comprising threaded and cylindrical implants having smooth alternatively blasted surfaces were made.

Study 1.

The study comprised 3 dogs (labrador) which each had 6 threaded fixtures implanted (i.e. 18 in all). Of these 18 fixtures 9 had a conventional structure of the surface whereas the remaining 9 fixtures had been treated with a blasting method in accordance with the above Example A.

After a healing period of 1 respectively 6 months for both implant surfaces a torque test was performed in which the fixtures were screwed out and the torque which was necessary for this was registered with a torque meter.

The results were that the fixtures having conventional surfaces could be removed when torques in the range of 35 - 45 Ncm were applied. The fixtures which had been blasted could not be removed although a torque of 100 Ncm was applied. The testing equipment broke at torques exceeding 100 Ncm.

Study 2.

In this animal study both cylindrical and threaded fixtures having alternatively conventional surfaces and surfaces blasted according to the Example were compared in accordance with the following schedule:

Cylindrical	implant,	conventional	surface:		6 fixtures
"	"	blasted	"	:	6 "
Threaded	"	conventional	"	:	6 "
"	"	blasted	"	:	6 "

The 24 fixtures were implanted in 6 dogs, 4 in each, in accordance with a random schedule.

The result was that all the fixtures having a conventional surface could be removed at torques ranging from 30 to 40

Ncm. The blasted implants, both the threaded and the cylindrical ones, could however not be removed despite the application of torques of 100 Ncm. The equipment could not stand higher torques.

The conclusion is that the blasted surface gives a considerably higher strength in the bond titanium - bone.

As mentioned in the introduction,it should be understood that the present invention only has been illustrated by means of dental implants and that the method according to the invention of course is applicable to all kinds of bone implants having a surface to be implanted made of titanium or titanium alloys.

It further should be pointed out that, although the invention has been illustrated using air blasting, airless (mechanical) blasting and wet blasting also can be used.

CLAIMS.

1. Method for treating the surface of implants of titanium or an alloy of titanium, c h a r a c t e r i z e d in that the surface is blasted with particles of a titanium oxide.

2. Method according to claim 1, c h a r a c t e r i z e d in that the oxide is titanium dioxide.

3. Method according to claim 1 or 2, c h a r a c t e r i z e d in that the particles used have a size within the range of 1 to 2000 µm.

4. Method according to claim 3, c h a r a c t e r i z e d in that the particles used have a size within the range of 1 to 300 µm.

5. Method according to claim 3, c h a r a c t e r i z e d in that the particles used have a size within the range of 1 to 50 µm.

6. Method according to any one of the preceding claims, c h a r a c t e r i z e d in that the implant treated is a dental implant.

7. Implant, c h a r a c t e r i z e d in that it has been treated according to any one of claims 1 - 6.

8. Method for treating toothlessness, c h a r a c t e r i z e d in that a dental implant is treated in accordance with any one of claims 1 - 6, in that one or several implants whose surface have been so treated is implanted into the jaw of a patient in need of such a treatment, whereafter one or several teeth or dental bridges are attached to the implant(s).

9. Method for increasing the fixation strength of orthopaedic implants c h a r a c t e r i z e d in that the surface of the implants is blasted with a titanium oxide in accordance with any one of claims 1 - 5.

INTERNATIONAL SEARCH REPORT

International Application No PCT/SE 91/00672

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC5: A 61 C 8/00, A 61 F 2/02, A 61 L 27/00

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
IPC5	A 61 C; A 61 F; A 61 L

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in Fields Searched[8]

SE,DK,FI,NO classes as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category *	Citation of Document,[11] with indication, where appropriate, of the relevant passages[12]	Relevant to Claim No.[13]
A	SE, B, 416175 (P I BRÅNEMARK ET AL) 8 December 1980, see the whole document --	1-7,9
A	SE, B, 462565 (PERMELEC ELECTRODE LTD) 16 July 1990, see the whole document --	1-7,9

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* Special categories of cited documents:[10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance, the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance, the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
9th January 1992	1992 -01- 15
International Searching Authority: SWEDISH PATENT OFFICE	Signature of Authorized Officer: Jack Hedlund

Form PCT/ISA/210 (second sheet) (January 1985)

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

V. ☒ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [1]

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☒ Claim numbers 8, because they relate to subject matter not required to be searched by this Authority, namely:

See PCT Rule 39.1(iv): Methods for treatment of the human or animal body by surgery or therapy as well as diagnostic methods.

2. ☐ Claim numbers....., because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claim numbers....., because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4(a).

VI. ☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [2]

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the the claims. It is covered by claim numbers:

4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

☐ The additional search fees were accompanied by applicant's protest.

☐ No protest accompanied the payment of additional search fees.

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.PCT/SE 91/00672

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the Swedish Patent Office EDP file on 31/10/91
The Swedish Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
SE-B- 416175	80-12-08	BE-A-	881953	80-06-16
		CA-A-	1157694	83-11-29
		CH-A-B-	653245	85-12-31
		DE-A-C-	3007446	80-09-18
		FR-A-B-	2450599	80-10-03
		GB-A-B-	2045083	80-10-29
		JP-A-	55120864	80-09-17
		LU-A-	82222	80-06-06
		NL-A-	8001241	80-09-09
		SE-A-	7902035	80-09-08
		US-A-	4330891	82-05-25
SE-B- 462565	90-07-16	DE-A-C-	3639607	87-05-27
		FR-A-	2591529	87-06-19
		GB-A-B-	2183256	87-06-03
		JP-C-	1575861	90-08-24
		JP-A-	62120403	87-06-01
		US-A-	5034186	91-07-23